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**(54) TREATMENT OF SILICON NITRIDE POWDER**

(11) 3-237008 (A) (43) 22.10.1991 (19) JP  
(21) Appl. No. 2-31005 (22) 9.2.1990  
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(51) Int. Cl<sup>5</sup>. C01B21/068, C04B35/58

**PURPOSE:** To improve the suitability of specified  $\text{Si}_3\text{N}_4$  powder to slurring and to considerably reduce the viscosity of a slurry of the  $\text{Si}_3\text{N}_4$  powder by heat-treating the  $\text{Si}_3\text{N}_4$  powder in gaseous  $\text{N}_2$  or in an inert atmosphere.

**CONSTITUTION:**  $\text{Si}_3\text{N}_4$  powder having  $\geq 6\text{m}^2/\text{g}$  BET specific surface area and 0.3-18wt.% oxygen content is heat-treated at 300-1,500°C for 1-24 hr in gaseous  $\text{N}_2$  or in an inert atmosphere to convert silanol groups in the  $\text{Si}_3\text{N}_4$  powder into silica as well as to volatilize and remove acid groups.

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**(54) HIGH PURITY PHOSPHORIC ACID AND ITS PRODUCTION**

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**PURPOSE:** To decrease amts. of various impurity metal elements by adding seed crystal to the raw phosphoric acid in an atmosphere purified by removing suspending fine particles so as to obtain phosphoric acid hemihydrate crystal, subjecting this hemihydrate to purification and then to micro filtration to remove suspending fine particles.

**CONSTITUTION:** Air is purified with using a high-performance filter to remove suspending fine particles so that the purified atmosphere contains 0-10000 fine particles of  $\geq 0.3\mu\text{m}$  size per  $\text{1ft}^3$ . In this atmosphere, the source phosphoric acid of  $\geq 70\text{wt.}\%$  concn. is kept at a temp. range lower than the saturation temp. by 0-10°C, to which the seed crystal is added by 0.1-2% of the mass of the raw phosphoric acid, and then the temp. is lowered to obtain acicular phosphoric acid hemihydrate crystal having 0.5-10mm major axis. Then this hemihydrate crystal is washed with high-purity water, maintained at a temp. higher than the saturation temp. by 0-10°C, purified by sweating, molten, and subjected to micro filtering so that the purified phosphoric acid contains suspending particles of  $\geq 0.5\mu\text{m}$  particle size by  $\leq 50$  particles per  $\text{1ml}$ . By this method, the obtd. high purity phosphoric acid contains impurities by  $\leq 20\text{ppb}$  Fe,  $\leq \text{ppb}$   $\mu\text{n}$ , and  $\leq 40\text{ppb}$  Na in terms of 85wt.%  $\text{H}_3\text{PO}_4$  concn.